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**SOLUBILITIES OF HIGH EXPLOSIVES: REMOVAL
OF HIGH EXPLOSIVE FILLERS FROM MUNITIONS
BY CHEMICAL DISSOLUTION**

Michael E. Sitzmann, et al

Naval Ordnance Laboratory
White Oak, Maryland

21 November 1973

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**SOLUBILITIES OF HIGH EXPLOSIVES
REMOVAL OF HIGH EXPLOSIVE FILLERS
FROM MUNITIONS BY CHEMICAL DISSOLUTION**

by

Michael E. Sitzmann
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ABSTRACT: In a search for a good overall solvent for high explosives, a systematic study of the solubilities of representative explosives in various single and binary solvents was made. Dimethylformamide (DMF) is judged to be a good overall solvent for explosives when factors such as the solubility values, physical properties, cost, availability, convenience and toxicity of the solvent are considered. DMF forms a solvate with HMX and is not useful as a solvent for HMX. A butyrolactone-dimethylsulfoxide mixture is recommended as a solvent for HMX. Sensitivities of slurries of explosives and solvents were determined. Studies were made to estimate the size of pump required to supply solvent for removal of explosives from munitions.

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A method for removal of high explosive fillers from ordnance items based on chemical dissolution would have several uses. A good overall solvent is required for this purpose. This report describes a study to find such a solvent. The sensitivities of slurries of explosives and solvents are also described. This work has been supported by Naval Explosive Ordnance Disposal Facility, Indian Head, Maryland, PO-3-0009.

ROBERT WILLIAMSON II

Carl Boyars
CARL BOYARS
By direction

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INTRODUCTION

Several methods are available for the removal of high explosive fillers from ordnance items. However, these methods (steaming, high pressure water jetting, burning and machining) are not always applicable to every situation. For example, certain new types of explosives are not readily melted or disintegrated by the application of wet steam. Use of the other methods to remove these types of explosives may present safety, equipment or ecological hazards. A situation may arise in which a piece of foreign ordnance cannot be defused. The weapon could be rendered safe by dissolving out the main charge with a solvent. Certain explosives are extremely sensitive, especially after degradation resulting from adverse storage. Solutions of these explosives would not be sensitive and could be removed safely from the ordnance items. Thus, a method for removal of high explosive fillers based on chemical dissolution would have several uses. A good overall solvent for high explosives would be desirable for this purpose. To find such a solvent, a systematic study of the solubilities of representative high explosives in various solvents was undertaken.

APPROACH

The solubilities of representative high explosives in various solvents at temperatures ranging from 25°C to 98°C were determined. Studies of the solubilities of various PBX's were also made. Single solvent systems as well as binary and ternary solvent mixtures were investigated in the search for a good overall solvent for high explosives. The sensitivities of several slurries of explosives and solvents were measured to insure that such mixtures would not be hazardous. Model "bombs" were prepared and tests were conducted to estimate the size of pump (pumping pressure and pumping rate) required to supply solvent to remove explosives from munitions. Such a pump has been purchased and further tests will be conducted with it.

RESULTS AND DISCUSSIONA. Determination of Solubilities

An excess of the explosive was stirred with the solvent for a 5 minute period. The mixture was filtered and the weight of insoluble explosive was measured. The amount of explosive dissolved in the solvent was taken as the difference between the amount of explosive added and the amount undissolved. Solubilities over a 30 minute period were also determined for a number of the explosives and solvents. Generally these 30 minute values were the same as those for the 5 minute period, (see Table 10). This would indicate that any additional explosive dissolved after the 5 minute period is very small and the 5 minute value is an accurate measure of the solubility of the explosive in the solvent. (See also experimental section.)

B. Representative High Explosives

The representative high explosives included standard explosives such as RDX, HMX, TNT and tetryl in addition to "exotic" explosives such as HNS, DATB, TATB and DIPAM. The "exotic" explosives are much less soluble than the standard explosives. They are not now in large scale use but are expected to be the explosives of the future in U.S. and foreign munitions which encounter high thermal environments.

C. Solubilities in Single Solvents

RDX and HMX represent two of the least soluble of the standard explosives. Solubility data on these two standard explosives along with solubility data on several of the "exotic" explosives were determined. The results are given in Tables 1 through 5.

Some of the data given in Tables 1 through 4 are shown in Table 6 in order to facilitate a comparison of the better solvents for the explosives under study. An examination of Table 6 reveals that RDX has about equal solubilities in dimethylsulfoxide (DMSO), dimethyl-formamide (DMF) and N-methylpyrrolidone (NMP). NMP appears to offer an advantage over DMSO and DMF as a solvent at 25°C for DATB and HNS. However, both NMP and DMF have one major disadvantage; that is, they form a solvate with HMX. Shortly after HMX is dissolved in NMP or DMF, precipitation of the solvate crystals occurs. This would make pumping of such a solution difficult and possibly hazardous as crystallization could occur in the pump. DMSO appears to be a very good solvent for HMX but the melting point of DMSO (18°C, 65°F) detracts from its usefulness. For practical purposes the solvent should remain liquid down to -20°F or ca-30°C. Butyrolactone (mp -42°C), the next best solvent for HMX, is a much poorer solvent for HMX than is DMSO.

D. Binary and Ternary Solvent Mixtures

A binary mixture of solvents may give better solvent properties than either of the two individual components. For example, the precipitation of the HMX solvate from NMP and DMF may be prevented if DMSO is added to each of these solvents. These DMSO mixtures with NMP and DMF should have a melting point of ca-30°C to meet the requirement that the solvent remain liquid down to this temperature. To determine the composition of a NMP-DMSO mixture that melts at -30°C, a phase diagram (Figure 1) was prepared. In the phase diagram the melting points of various NMP-DMSO mixtures are plotted versus the mole percent composition of the mixture. The melting point of each mixture was determined by freezing the mixture and then slowly allowing it to warm past the melting point. The temperature of the mixture was plotted versus the time. The melting point of the mixture was taken as the point where the break in the curve was obtained. A break in the time-temperature curve is expected at the melting point since the temperature of the mixture rises at a faster rate once all the frozen material is melted. The melting points obtained in this manner were confirmed visually by noting the temperature at which the material appeared to be completely melted. Figure 1 shows that a mixture of NMP and DMSO melting at -30°C must be composed of ca 75 mole% NMP and 25 mole% DMSO. This corresponds to 79.2 % NMP and 20.8 % DMSO by weight. Figures 2 and 3 show the phase diagrams for mixtures of DMSO with DMF and butyrolactone. A DMF-DMSO mixture melting at -30°C must be ca 73 mole% DMF and 27 mole% DMSO corresponding to 71.7% DMF and 28.3% DMSO by weight. A butyrolactone-DMSO mixture composed of 71 mole% butyrolactone and 29 mole% DMSO (73.0% butyrolactone and 27.0% by weight) melts at -30°C.

The melting point of a ternary mixture (16.4 mole% DMF, 13.1 mole% NMP and 70.5 mole% DMSO) was determined in order to compare it with the melting points of binary mixtures containing the same mole percentage of DMSO. The melting point (3.7°C) of the ternary mixture is approximately the same as that of a binary mixture of 29.5 mole% DMF and 70.5 mole% DMSO or a binary mixture of 29.5 mole% NMP and 70.5 mole% DMSO. Thus it does not appear that a ternary mixture would offer any advantage over a binary mixture as far as the melting point is concerned.

Solubilities of representative high explosives in several binary solvent mixtures are given in Table 7. Many of the explosives have about the same solubility in a particular binary solvent mixture as in the largest component of the mixture. For example, HNS has a solubility value of 2.4 g/100 g in NMP; the solubility value for HNS in the NMP-DMSO mixture is 2.3 g/100 g. It was hoped that the addition of DMSO to NMP and DMF would prevent precipitation of the HMX solvate from these solvents. Unfortunately, this was not the case. The

precipitation of the HMX solvate from these binary mixtures makes them unsuitable as solvents for HMX. The best solvent for HMX that will remain liquid down to ca-30°C is the butyrolactone-DMSO mixture (73/27). HMX has a solubility of 26 g/100g in this solvent. Thus, for HMX, a binary solvent mixture offers an advantage over all of the single solvents. However, for most other high explosives the convenience of a single solvent system overrides any advantages offered by the binary solvent mixtures.

E. Choice of a Good Overall Solvent for High Explosives

DMSO, NMP and DMF are the better single solvents for high explosives. However, the requirement that the solvent remain liquid down to -30°C prevents the use of DMSO (mp 18°C) or NMP (mp -21°C) in a single solvent system. This would leave DMF (mp -60°C) as the best single solvent for high explosives. The convenience of a single solvent system must be weighed against any advantages offered by binary solvent mixtures. Only for HMX does a binary solvent mixture offer a large advantage over a single solvent system. DMF forms a solvate with HMX and thus cannot be used as a solvent for HMX.

Considering all the factors in the choice of a solvent (solubility values, physical properties, cost, availability, convenience and toxicity), DMF was judged to be a good overall solvent for high explosives. However, since DMF forms a solvate with HMX, the butyrolactone-DMSO mixture (73/27) is the solvent of choice for HMX. Solubilities of explosives in DMF at various temperatures are shown in Table 8. Solubilities of HMX in the butyrolactone-DMSO mixture at various temperatures are given in Table 9. Some solubility values at temperatures less than 25°C were determined in order to have information available in case the solvents were used in cold environments.

F. Solubilities of PBX's

PBX's such as PBXN-103, PBXN -5 and PBXN -4 which do not contain cross-linked polymers can be dissolved and/or disintegrated with solvents. For PBX's which contain a large proportion of HMX, the butyrolactone-DMSO mixture (73/27) would be the solvent of choice; otherwise, DMF can be employed. However, no solvent could be found to disintegrate a PBX containing cast-cured cross-linked polymers (PBXN-101 and PBXN-106).

G. Rates of Solution

A comparison of the rates of solution of each explosive in various solvents was desired. This objective was thought to be possible by taking standard size pressed pellets of the explosives and measuring the times to complete solution in a variety of solvents under fixed

conditions (fixed rate of stirring, temperature, etc.). However, the pellets crumbled under the influence of the solvent and no measurement of rates could be accomplished.

II. Sensitivities of Slurries of Explosives and Solvents

It is likely that slurries of explosives are less sensitive than the explosives by themselves. Hence, a method to remove high explosive fillers by dissolving would be expected to be relatively safe in comparison to most other methods available for removal of these fillers. In order to insure that this is the case, the NOL large scale gap test was used as a measure of the sensitivities of several slurries of explosives and solvents. The test apparatus was set in a position such that any explosive that settled from the slurry would be in close proximity to the booster charge. This configuration should insure maximum sensitivity of the slurry.

A slurry of TNT and DMF containing a 50% excess of TNT over the amount of TNT required to give a saturated solution in DMF at 25°C (68% TNT by weight) failed to give a positive test at 50 cards. For reference, TNT at 65.0% of the theoretical maximum density (TMD) has a value of 282 cards. Other card values for TNT are 224 at 80.6% TMD, 193 at 95.7% TMD and 133 at 96.6% TMD.

A mixture of RDX and DMF containing a 50% excess of RDX over the amount of RDX required to give a saturated solution in DMF at 25°C (35% RDX by weight) did not give a positive test at 50 cards. A mixture of RDX and DMF (67% RDX by weight) gave a positive test at 100 cards but failed at 150 cards. Values for RDX are 336 cards at 85.1% TMD and 323 cards at 91.0% TMD.

A slurry of HMX and butyrolactone-DMSO (73/27) that contained 67% HMX by weight gave a negative test at 150 cards as did a slurry of ammonium perchlorate and DMF (67% ammonium perchlorate by weight). For reference, HMX shows approximately the same gap test sensitivity as RDX. The oxidizing agent (ammonium perchlorate) when mixed with a fuel (DMF) in a ratio of 67/33 does not give a very sensitive mixture. Mixtures richer in ammonium perchlorate may be more sensitive but such concentrations should never be attained in a practical situation.

The above tests indicate that slurries of explosives are less hazardous than the explosives by themselves and a method to remove high explosive fillers by dissolving would be relatively safe in comparison to most other methods available for removal of these fillers.

I. Estimation of Pump Size Required to Supply Solvent for Removal of Explosives from Munitions

It was desired to estimate the pumping pressure (psi) and pumping rate (gal/min) that would be required to efficiently remove explosive

from an ordnance item. If sufficient pumping pressure is used, a portion of the explosive may be flushed out of the ordnance item without being dissolved in the solvent. Some explosives may contain components (aluminum, for example) which will not dissolve in the organic solvents employed. Insoluble components of explosives would have to be removed from the ordnance item by the flushing action of the solvent. A filter system would remove any undissolved materials from the solvent so that the solvent could be recycled through the pump.

In order to estimate the pumping pressure and pumping rate required to remove explosives from ordnance items, studies on model "bombs" were conducted. Several charges of TNT bearing explosives (HBX-1 and composition B) were cast into glass tubes 2.75 inches in diameter. These model "bombs" had a hole in the side about 1 inch in diameter for the introduction of solvent. The solvent was impinged on the explosive by forcing the solvent from a pressure tank through a nozzle with air pressure. The flow rates were varied by changing air pressure and nozzle sizes. Experiments were performed to determine how efficiently the explosive in the model "bombs" could be penetrated with solvents. For example, using DMF as a solvent (at ambient temperature, 32°C) with a 0.074 inch nozzle and an air pressure of 33 psi, the 2.75 inches of explosive in the model "bomb" was penetrated in 2 minutes. Approximately 1.5 gal of DMF was required. From the experiments, it was estimated that a pump capable of producing 10 gal/min at 60 psi would be more than adequate to supply solvent to remove explosives from ordnance items. A system that utilizes such a pump will be developed and tested as a method to remove high explosives from munitions. The system will be designed so that the solvent can be filtered and recycled.

SUMMARY AND CONCLUSIONS

A method for removal of high explosive fillers from ordnance items based on chemical dissolution would have several uses. A good overall solvent for high explosives is required for this purpose. In the search for a good overall solvent for high explosives, single solvent systems as well as binary and ternary solvent mixtures were investigated. Considering all the factors in the choice of a solvent (solubility values, physical properties, cost, availability, convenience and toxicity), dimethylformamide was judged to be a good overall solvent for high explosives. However, dimethylformamide forms a solvate with HMX and cannot be used as a solvent for HMX. A butyrolactone-dimethylsulfoxide mixture (73/27) is the solvent of choice for HMX. The gap test sensitivities of several slurries of explosives and solvents indicated that slurries of explosives are less sensitive than the explosives by themselves. Hence, a method to remove high explosive fillers by dissolving is relatively safe compared to most

other methods available for removal of these fillers. From experiments with model "bombs", it was estimated that a pump capable of producing 10 gal/min at 60 psi would be more than adequate to supply solvent to remove explosives from ordnance items. A system utilizing such a pump will be developed and tested as a method for removal of high explosives from munitions.

EXPERIMENTAL

A. Solvents

The solvents purchased for this study were used as received. N-methylpyrrolidone, dimethylacetamide and aniline were practical grade solvents. All other solvents were of reagent grade quality.

B. Solubility Determinations

A volume of 10, 20, or 50 ml of solvent was employed depending on the solubility of the explosive in the solvent. The larger volumes were used for the less soluble explosives. A volume of solvent (measured by pipet) was weighed and stirred at the desired temperature in a water bath. An excess of explosive was added to the solvent and the mixture was stirred for a 5 minute period. The mixture was filtered (a heated filter funnel was used for filtration of mixtures above 25°C) and the insoluble explosive was pulled dry with suction and then washed with water (the explosives with an appreciable water solubility were insoluble in either carbon tetrachloride or chloroform and were washed with one of these solvents). The insoluble explosive was dried and weighed. The weight of explosive dissolved in the solvent was taken as the difference between the amount of explosive added to the solvent and the amount of insoluble explosive. The solubilities were calculated as grams of explosive per 100 grams of solvent.

TABLE 1

Solubility of RDX (g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>25°C</u>	<u>40°C</u>	<u>60°C</u>	<u>80°C</u>	<u>98°C</u>
Dimethylsulfoxide	41	51	66	87	113
Dimethylformamide	37	45	58	76	96
N-Methylpyrrolidone	40	47	58	72	84
Butyrolactone	14		28	41	61
Dimethylacetamide	33				
Acetone	8.2	12	17 ^a		
Cyclohexanone	7.7 ^b		15	23	33
Acetonitrile	5.5 ^b	8.8	14	26 ^a	
Cyclopentanone					38 ^c
Hexamethylphosphoramide	16				45
Aniline	1.9				

a. At the boiling point of the solvent.

b. In general, our solubility data were in agreement with values that had been previously reported in the literature. However, there appears to be a discrepancy between our data and that given in the Engineering Design Handbook¹ for the solubility of RDX in acetonitrile and cyclohexanone. The handbook gives values of 11.3 and 13.0 g/100 g, respectively, for the solubility of RDX in acetonitrile and cyclohexanone at 28°C. To be consistent with our other data we obtained solubility values at 25°C after a 5 minute period for RDX in acetonitrile (5.5g/100g) and cyclohexanone (7.7g/100g). The value for RDX in acetonitrile at 25°C was checked several times using different amounts of solvent and in one instance a different brand of solvent. The values obtained were 5.4, 5.5, 5.6, and 5.5g/100g. After a 30 minute period, a value of 5.5g/100g was obtained. Duplicate experiments for the solubility of RDX in cyclohexanone at 25°C gave 7.7g/100g and 7.6g/100g.

c. After a 30 minute period at 94°C.

TABLE 2

Solubility of HMX (g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>25°C</u>	<u>60°C</u>	<u>80°C</u>	<u>98°C</u>
Dimethylsulfoxide	57	68		89
Dimethylformamide	Solvate ^a			
N-Methylpyrrolidone	Solvate ^a			
Dimethylacetamide	Solvate ^a			
Butyrolactone	12	20		35
Acetone	2.8	4.2 ^b		
Acetonitrile	2.0		7.3 ^b	
Cyclohexanone	1.0			8.9
Hexamethylphosphoramide	1.4			37 ^c

a. Shortly after the HMX dissolves, precipitation of the solvate crystals occurs.

b. At the boiling point of the solvent.

c. After a 30 minute period.

TABLE 3

Solubility of HNS (g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>25°C</u>	<u>60°C</u>	<u>80°C</u>	<u>98°C</u>
Dimethylsulfoxide	1.4	2.4	3.6	9.1
Dimethylformamide	1.5	3.2	4.6	7.0
N-Methylpyrrolidone	2.4	4.6	6.4	8.4
Butyrolactone	0.4			3.2
Dimethylacetamide	1.2			5.4

(Table 3 continued next page)

TABLE 3 (Cont.)

Solubility of HNS (g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>25°C</u>	<u>60°C</u>	<u>80°C</u>	<u>98°C</u>
Acetone	<0.1	0.4 ^a		
Nitrobenzene	<0.1			0.8
Hexamethylphosphoramide				3.3
Cyclohexanone				1.1

a. At the boiling point of the solvent.

TABLE 4

Solubility of DATB (g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>25°C</u>	<u>60°C</u>	<u>80°C</u>	<u>98°C</u>
Dimethylsulfoxide	2.2			19
Dimethylformamide	2.5	5.1	9.7	14
N-Methylpyrrolidone	4.5			17
Butyrolactone	0.8			4.8
Acetone	0.4	1.4 ^a		
Aniline	2.2			
Trifluoroacetic Acid	<0.1			

a. At the boiling point of the solvent.

TABLE 5

Solubility of TATB (g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>25°C</u>	<u>98°C</u>
Dimethylsulfoxide	<0.1	0.5
N-Methylpyrrolidone	<0.1	0.8
Aniline	<0.1	
Sulfuric Acid	14	18
Trifluoroacetic Acid	<0.1	

TABLE 6

Solubilities of Explosives in the Better Single Solvents
(g/100 g solvent after 5 min. stirring)

<u>Solvent</u>	<u>RDX</u>		<u>HMX</u>		<u>HNS</u>		<u>DATB</u>	
	<u>25°C</u>	<u>98°C</u>	<u>25°C</u>	<u>98°C</u>	<u>25°C</u>	<u>98°C</u>	<u>25°C</u>	<u>98°C</u>
DMSO	41	113	57	89	1.4	9.1	2.2	19
DMF	37	96	Solvate ^a		1.5	7.0	2.5	14
NMP	40	84	Solvate ^a		2.4	8.4	4.5	17
Butyro- lactone	14	61	12	35	0.4	3.2	0.8	4.8

a. Shortly after the HMX dissolves, precipitation of the solvate crystals occurs.

TABLE 7
Solubilities of Explosives in Binary Solvent Mixtures^a at 25°C
(g/100 g of solvent after 5 min. stirring)

<u>Solvent</u>	<u>m.p. of Solvent</u>	<u>RDX</u>	<u>HMX</u>	<u>TNT</u>	<u>HNS</u>	<u>DATB</u>	<u>Ammonium Picrate</u>	<u>Nitro-Guanidine</u>
DMSO	18	41	57	128	1.4	2.2	75	33
NMP	-21	40	Solvate ^b	118	2.4	4.5	62	21
NMP 79.2 ^a	-30	41	Solvate ^b	120	2.3	4.6	70	24
DMSO 20.8 ^a								
DMF	-60	37	Solvate ^b	142	1.5	2.5	90	15
DMF 71.7 ^a	-30	39	Solvate ^b	142	1.7	2.9	95	21
DMSO 28.3 ^a								
Butyro-lactone	-42	14	12		0.4	0.8		
Butyro-lactone	73.0 ^a	-30		26				
DMSO	27.0 ^a							

a. Solubilities in single solvents are included for comparison.

b. Shortly after the HMX dissolves, precipitation of the solvate crystals occurs.

TABLE 8

Solubilities of Explosives in DMF
(g/100 g of solvent after 5 min. stirring)

<u>Explosive</u>	<u>0°C</u>	<u>15°C</u>	<u>25°C</u>	<u>60°C</u>	<u>80°C</u>	<u>98°C</u>
RDX	27		37	58	76	96
TNT	90	119	142			
HNS			1.5	3.2	4.6	7.0
DATB			2.5	5.1	9.7	14
Ammonium Picrate	84		90			
Nitro- guanidine			15	20		28
Ammonium nitrate	47		54			
Ammonium Perchlorate	46		47			
DIPAM			>27 ^a			
Picric Acid			>225 ^b			

a. Difficulty in getting consistent results for the solubility of DIPAM in DMF was experienced. Concentrated solutions of DIPAM in DMF are very viscous and this may account for the difficulty. However, we are confident that the solubility of DIPAM in DMF at 25°C is at least 27 g/100 g.

b. An amount of 21.0 g of picric acid was added in portions to 9.34 g of DMF stirred at 25°C. After 5 minutes all the solid appeared to be dissolved giving a very viscous solution. The solubility value appears to be greater than 225 g/100 g.

TABLE 9

Solubility of HMX in Butyrolactone-DMSO (73/27)
(g/100 g of solvent after 5 min. stirring)

<u>0°C</u>	<u>25°C</u>	<u>60°C</u>	<u>98°C</u>
20	26	33	49

TABLE 10

Comparison of Solubilities (g/100 g)
after 5 and 30 min. stirring at 25°C

<u>Explosive</u>	<u>Solvent</u>	<u>5 min.</u>	<u>30 min.</u>
DATB	Dimethylsulfoxide	2.2	2.3
HNS	Dimethylsulfoxide	1.4	1.4
RDX	Dimethylsulfoxide	41	41
RDX	Dimethylformamide	37	36
RDX	Acetonitrile	5.5	5.5
HMX	Dimethylsulfoxide	57	58
HMX	Butyrolactone	12	13

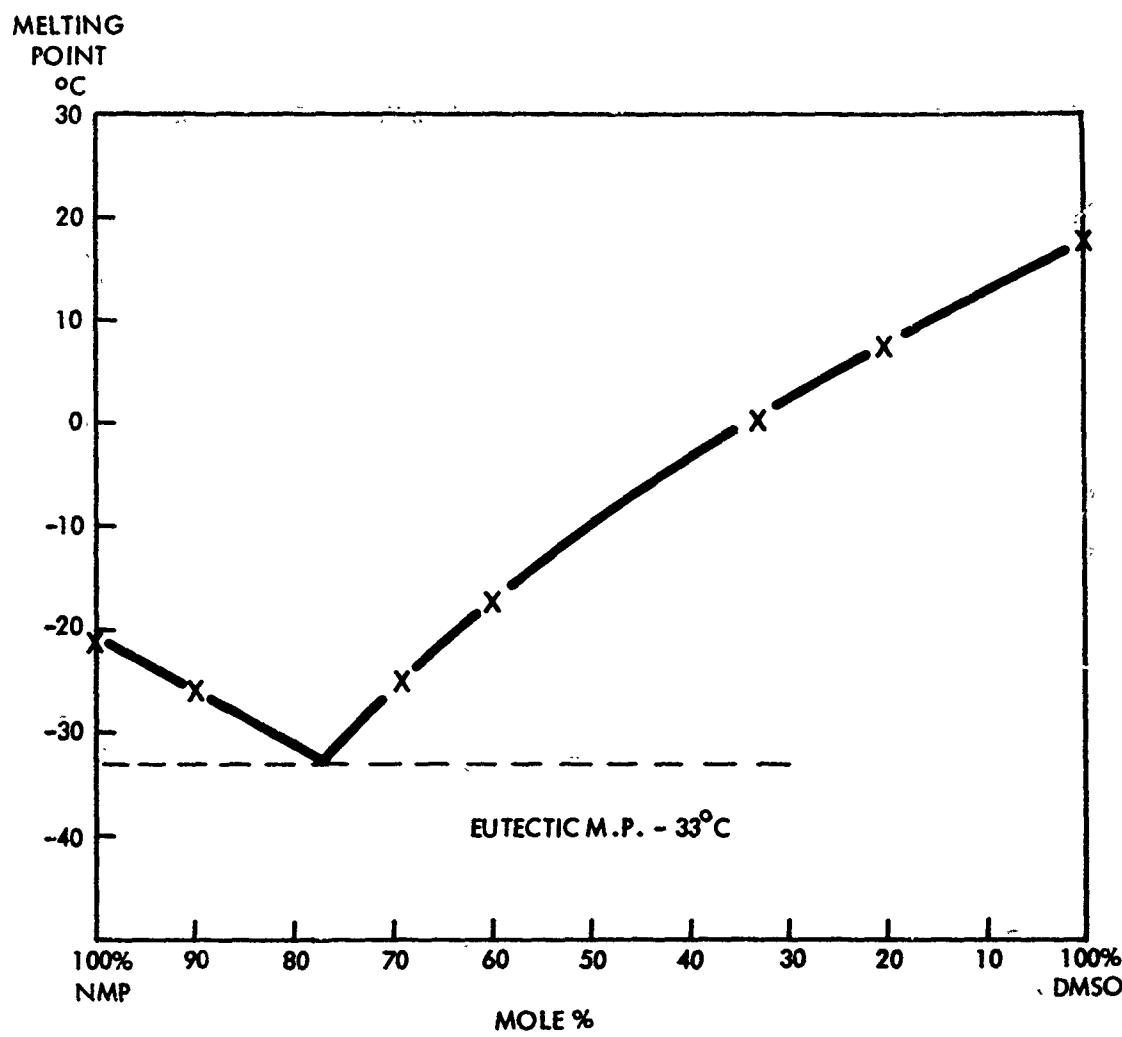


FIG. 1 (U) MELTING POINTS OF NMP - DMSO MIXTURES (U)

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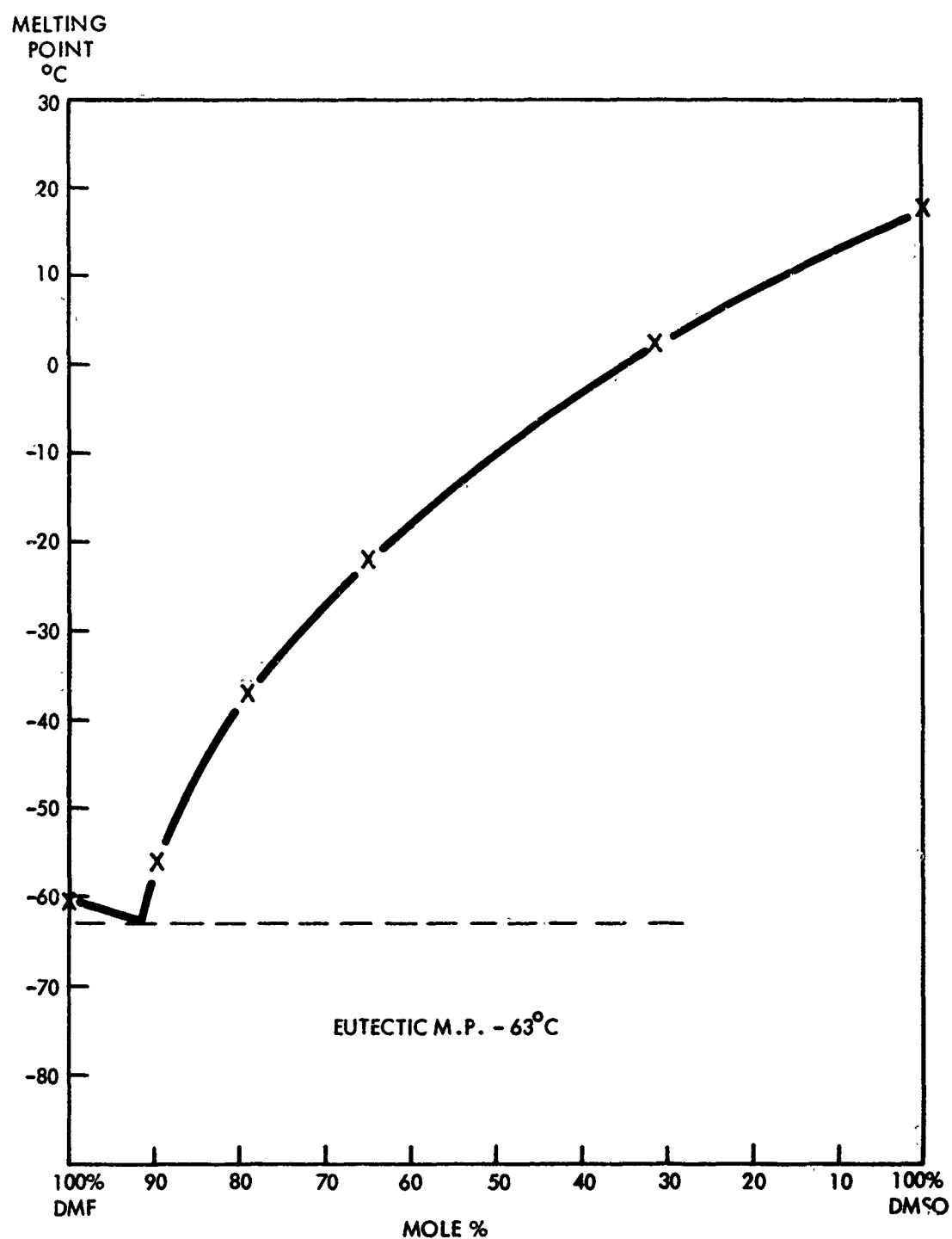


FIG. 2 (U) MELTING POINTS OF DMF - DMSO MIXTURES (U)

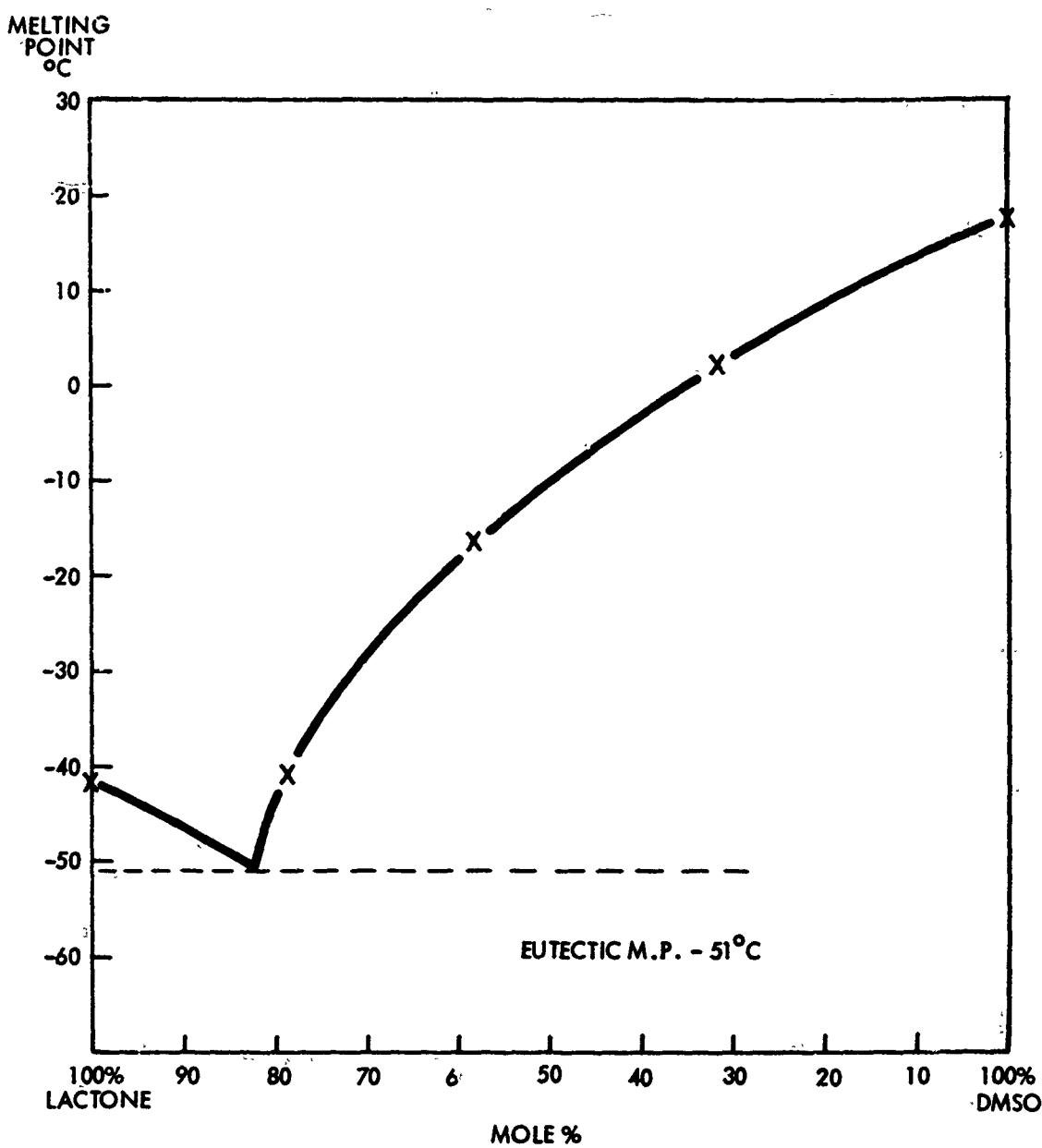


FIG. 3 (U) MELTING POINTS OF BUTYROLACTONE - DMSO MIXTURES (U)

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